

# PATENT SPECIFICATION

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## COMPLETE SPECIFICATION

### Heat Treatment of Steels Micro-alloyed with Nb/Ta and/or V

We, TRAFIKAKTIEBOLAGET GRÄNGESBERG-OXELOSUND, of 18 Gustaf Adolfs Torg, Stockholm, Sweden, a Swedish joint-stock company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

THIS INVENTION relates to a heat-treatment of steels micro-alloyed with Nb/Ta and/or V. The invention relates further to welded constructional elements manufactured of steel which is heat-treated according to the invention, or which in their entirety are subjected to such heat-treatment.

The term "micro-alloy steels" is to be understood to cover steels to which very small amounts of alloying elements of strong action are added, by which elements the properties of the base alloy are radically changed. Such steels, thus, may be defined as micro-alloy carbon steel, micro-alloy manganese steel, micro-alloy molybdenum steel, etc., the micro-alloying addition in such steels usually being at least ten times less than the remaining alloying contents in the base alloy.

This invention consists in heat treatment of steels micro-alloyed with Nb/Ta and/or V, wherein a steel containing Nb/Ta up to 0,06% and/or V up to 0,12%, but containing a total of Nb/Ta and V at least 0,004%, is solution treated at a temperature above 950°C, suitably below 1200°C and preferably between 1050°C and 1100°C, to be followed by a quenching, and that thereafter the steel is

tempered at between 450°C and 750°C, preferably at between 600° and 650°C, and wherein the steel contains in addition 0,08 to 0,3, preferably 0,10 to 0,25% carbon, 0,3 to 1,7, preferably 0,8 to 1,5% manganese, 0,01 to 0,5, preferably 0,02 to 0,5% silicon and at maximum 0,015% nitrogen and the steel contains at least 94, preferably 95% of iron.

The quenching is carried out, for example in oil or water or another quenching agent with a similar quenching effect, in such a manner, that substantially a martensitic structure is obtained. In order to facilitate the formation of martensitic structure in greater material thicknesses, for example over 20 mm, preferably alloying elements are added which are adapted to lower the critical cooling velocity. As examples of such alloying elements may be mentioned: chromium, nickel, molybdenum, copper, manganese and vanadium. The additions used for promoting martensite formation include one or more of these elements and amount to a total content of 2%. The amount of vanadium is up to 0.12% and manganese 0.3 to 1.7%.

The steel used in carrying out the invention contains at least 94% of iron, preferably 95%.

The steel to be treated according to the invention is previously known in rolled and normalized state. Said steel is characterised by good weldability in connection with increased strength. The increase in its yield strength which can be ascribed to niobium and/or vanadium, can amount to from 10 to 12 kg/mm<sup>2</sup>.

[Price 4s. 6d.]

The heat-treatment according to the invention results in a considerably higher strength increase, an increase in the yield strength-ultimate strength ratio, an improved notched bar impact strength over that in rolled state, and in maintained good weldability.

The strength properties achieved by the heat-treatment according to the invention are fully comparable to the properties shown by considerably more expensive, high-strength, low-alloy steels, for example the US steel "T1" which contains 0,15% carbon, 0,80% manganese, 0,25% silicon, 0,60% chromium, 0,32% copper, 0,50% molybdenum, 0,85% nickel, 0,06% vanadium and small additions of boron. This steel which is heat-treated by hardening from 900°C and tempering to between 620 and 690°C shows a yield strength of about 70 kg/mm<sup>2</sup>.

The steel heat-treated according to the invention is, however, considerably simpler and cheaper to produce and shows, above all, superiority in weldability. Due to the lower content of alloying elements, the steel shows less tendency of crack formation during the welding. As a result thereof, the steel heat-treated according to the invention can be cold-welded up to considerably greater material dimensions than low-alloy steels of the same strength.

The said good strength properties effected by the heat-treatment according to the invention are due to the fact, that niobium and/or vanadium, which are maintained in a solid solution during the quenching, are precipitated during the tempering as carbides in a very finely distributed state and cause what is usually called precipitation hardening. Niobium, among other elements, was previously known to have such an effect, but it was not known that full precipitation hardening is obtained at such low contents, rendering it possible to obtain extraordinary weldability in spite of the very high strength.

Both the solution temperature and the tempering temperature are of great importance for obtaining optimum results. The micro-alloying elements niobium and/or vanadium are caused to dissolve by the solution treatment. The accurate solution temperature depends to a certain extent on the composition, as will be discussed below. But it is true of all compositions that the desired advantages cannot be fully utilized when a solution temperature below 950° is chosen. Temperatures above 1200°C, on the other hand, do not bring about considerable additional improvements of the properties, but result in disadvantageous surface decarburization and scale formation. The said upper limit of 1200°C, however, cannot be said to be critical, because the disadvantages caused can be eliminated by a subsequent surface treatment and also by a treatment in protective gas. The said limit, thus, is set for practical reasons. The

preferred solution temperature lies for most compositions within the range from 1050 to 1100°C.

The object of the quenching is to maintain the micro-alloying elements niobium and/or vanadium in a supersaturated solid solution and to obtain a substantially martensitic structure. The quenching agent is to be chosen according to the material dimension in question. As a rule, water may be used, but oil may be suitable, particularly in the case of thinner material dimensions. When the material is of such thickness, for example above 20 mm, that under these conditions through hardening cannot be obtained, according to a modification of the invention such alloying elements may be added to the steel which promote martensite formation by lowering the critical cooling velocity. A substantially martensitic structure is desired in order to obtain by the subsequent precipitation process during the tempering a structure which is the most favourable with respect to the notched-bar impact strength.

After the micro-alloying elements are fixed in supersaturated solid solution, the said elements are precipitated in the form of carbides by tempering at between 450 and 750°C. When the tempering is carried out at a temperature below 450°C, no appreciable improvements of the strength properties are obtained, because carbides are not precipitated unless for practical reasons unreasonably long tempering periods are used. By tempering, on the other hand, at a temperature above 700°C, a build up of the precipitated particles is obtained which may be said to abolish the advantages gained by the finely distributed carbide precipitation. It is found most advantageous to choose a tempering temperature within the range from 575 to 625°C. When choosing the tempering temperature, it must also be observed that the temperature should be equal to or higher than the temperature at which the stress-relieving annealing is carried out.

As mentioned above, the accurate solution temperature depends to a certain extent on the composition. The solubility of niobium can be expressed by the equation

$$\log (\% \text{Nb}) + \log (\% \text{C}) = A - \frac{a}{T}$$

wherein A and a are constants found to be -0,63 and 2500 respectively, and where T = absolute temperature in degrees Kelvin. This equation shows that at a given composition there is no reason to choose a higher value of T than is obtained from the above equation, because at this temperature the entire niobium content is dissolved. For the aforesaid practical reasons, however, in many cases a lower solution temperature than this is chosen. If, on the other hand, a certain definite carbon content is desired and the operation should

occur at a certain definite solution temperature  $T$ , the equation teaches which minimum content of niobium must be added to render possible full utilization of the precipitation effect during the subsequent tempering. As an example may be mentioned that for a steel containing 0,030% niobium and 0,11% carbon  $T=1350^{\circ}\text{K.}=1077^{\circ}\text{C}$  is obtained. If, on the other hand, the problem was such, that a solution temperature of, for example,  $1000^{\circ}\text{C}$  should not be exceeded and that the carbon content desired was 0,11, one finds that 0,023% niobium is dissolved. The niobium amount added which exceeds this content, will not be dissolved unless the temperature is raised. Corresponding ratios apply also to vanadium, the values of the constants being different from those indicated in the above equation.

The invention will be disclosed more precisely in the following, reference being had to the accompanying drawings, whereof Figure 1 is a diagram showing the dependence of the hardness increase on the tempering temperature for different solution temperatures, Figure 2 shows the dependence of yield strength and ultimate strength on the solution temperature, Figure 3 shows the dependence of the notched-bar impact strength on the solution temperature, Figure 4 shows the influence of the tempering temperature on the yield strength for different solution temperatures, and Figure 5 shows the influence of the tempering temperature on the ultimate strength for different solution temperatures.

For obtaining the diagram according to Figure 1 platelets ( $10 \times 10 \times 2$  mm) were prepared of a niobium micro-alloy steel containing 0,11% carbon and 0,036% niobium. The platelets were solution treated for 15 minutes at different temperatures between  $900^{\circ}\text{C}$  and  $1300^{\circ}\text{C}$ , and then quenched and tempered for 15 minutes between  $450^{\circ}\text{C}$  and  $900^{\circ}\text{C}$ . Samples taken from the same charge but without niobium addition were treated in the same way. The hardness was determined, and the difference in hardness between the unalloyed steel and the niobium micro-alloy steel was calculated and plotted in the diagram as a function of the tempering temperature. The values originating from the same solution temperature were connected. As appears, the precipitation effect reached a maximum at  $650^{\circ}\text{C}$ . Due to a certain build up of the precipitated particles at higher temperatures, the hardness increase decreased at temperatures over  $650^{\circ}\text{C}$ . The hardness increase increased with rises of solution temperature up to  $1200^{\circ}\text{C}$ . At  $1300^{\circ}\text{C}$  an improvement in hardness increase could be observed in some cases, but the improvement was so insignificant that it did not compensate for the disadvantage by the higher temperature.

Figure 2 shows the relation between the yield-ultimate strength and the solution

temperature at a certain tempering temperature, in this case at  $600^{\circ}\text{C}$  for 30 minutes and cooling in air. The steel used contained 0,21% carbon, 0,04% silicon, 1,32% manganese, 0,20% phosphorus, 0,013% sulphur and 0,015% niobium. For reason of comparison it may be mentioned, that corresponding steel without niobium addition has a yield strength of about  $32 \text{ kg/mm}^2$  and an ultimate strength of about  $52 \text{ kg/mm}^2$ . As appears from the diagram, for a steel of this comparison no appreciable improvement at solution temperatures exceeding  $1100^{\circ}\text{C}$  is obtained.

Figure 3 shows the relation between the notched-bar impact strength and the solution temperature. The notched-bar impact strength was determined according to Charpy V. The notched-bar impact strength was expressed as the transition temperature related to an energy absorption of  $2,8 \text{ kgm}$  per square centimetre. Corresponding steels without niobium addition show a transition temperature at  $-15^{\circ}\text{C}$ .

Figure 4 shows the relation between the yield strength and solution temperature for different tempering temperatures in this case for 500, 600, 700 and  $750^{\circ}\text{C}$ . The tested steel has a composition of 0,22% carbon, 0,22% silicon, 1,32% manganese, 0,014% phosphorus, 0,012% sulphur and 0,022% niobium. As appears from the diagram, the strength increases at increased solution temperature. At  $900^{\circ}\text{C}$  the strength increase which can be directly related to niobium precipitation, is still small, because niobium probably is dissolved only to a very small extent. It is difficult to make an accurate statement on how large a portion of the strength increase is to be ascribed to the niobium precipitation at lower solution temperatures, but at higher solution temperatures this portion amounts to about  $25 \text{ kg/mm}^2$  for the yield strength. The increase in strength is greater at tempering to  $500^{\circ}\text{C}$  than at tempering to  $600^{\circ}\text{C}$ , but decreases thereafter to half of its value at  $700^{\circ}\text{C}$  and is 0 at  $750^{\circ}\text{C}$ . The said decrease is entirely due to the build up of precipitated niobium carbide particles.

Figure 5 shows the ultimate strength as a function of the solution temperature for different tempering temperatures. The conditions are substantially the same as in the tests according to Figure 4. The maximum strength increase is about  $30 \text{ kg/mm}^2$  for the ultimate strength.

#### WHAT WE CLAIM IS:—

1. Heat treatment of steels micro-alloyed with Nb/Ta and/or V, wherein a steel containing Nb/Ta up to 0,06% and/or V up to 0,12%, but containing a total of Nb/Ta and V at least 0,004%, is solution treated at a temperature above  $950^{\circ}\text{C}$ , suitably below  $1200^{\circ}\text{C}$  and preferably between  $1050^{\circ}\text{C}$  and  $1100^{\circ}\text{C}$ , to be followed by a quenching, and that thereafter the steel is tempered at between

- 450°C and 750°C, preferably at between 600° and 650°C, and wherein the steel contains in addition 0,08 to 0,30, preferably 0,10 to 0,25% carbon, 0,3 to 1,7, preferably 0,8 to 1,5% manganese, 0,01 to 0,5, preferably 0,02 to 0,5% silicon and at maximum 0,015% nitrogen and the steel contains at least 94, preferably 95% of iron.
- 5 2. Heat treatment according to claim 1, characterised in that the quenching is carried out in such a manner, that a substantially martensitic structure is obtained.
- 10 3. Heat treatment according to claim 2, characterised in that the quenching is carried out in oil or water or in another quenching agent with a similar quenching effect.
- 15 4. Heat treatment according to claim 2, characterised in that for facilitating the formation of martensitic structure at larger material dimensions, for example exceeding 20 mm, alloying elements are added which are adapted to lower the critical cooling velocity.
- 20 5. Heat treatment according to claim 3 or claim 4 characterised in that for lowering the critical cooling velocity chromium is added to the steel.
- 25 6. Heat treatment according to claim 4 or claim 5 characterised in that for lowering the critical cooling velocity nickel is added to the steel. 30
7. Heat treatment according to claim 4 or claim 5 characterised in that for lowering the critical cooling velocity molybdenum is added to the steel.
8. Heat treatment according to claim 4 or claim 5 characterised in that for lowering the critical cooling velocity copper is added to the steel. 35
9. Heat treatment according to any one of claims 4 to 8, characterised in that the alloying additions are a mixture of two or more of the additions according to claims 4—8. 40
10. Steel heat treated according to any of the claims 1—9.
11. Welded constructional element manufactured of steel according to claim 10. 45

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3 SHEETS

COMPLETE SPECIFICATION  
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SHEET 1

FIG. 1

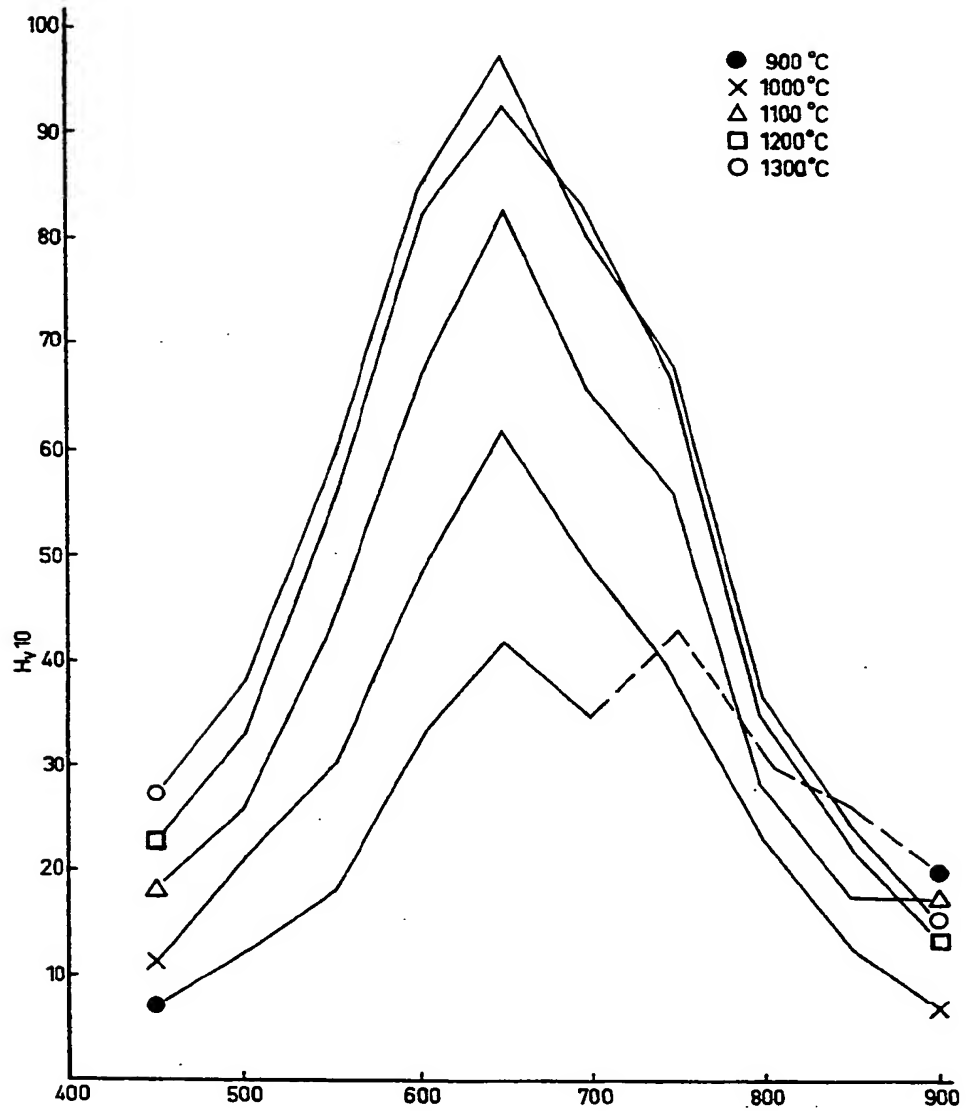


FIG. 2

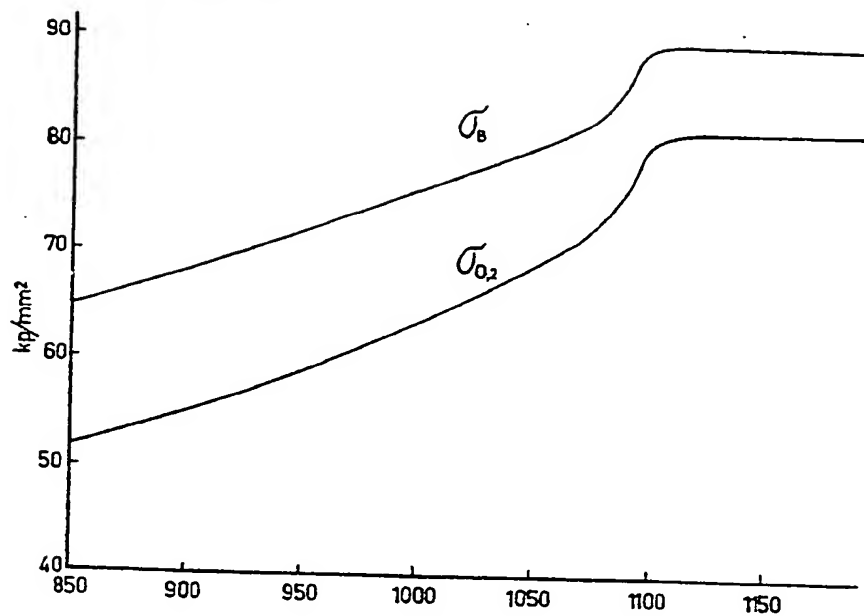
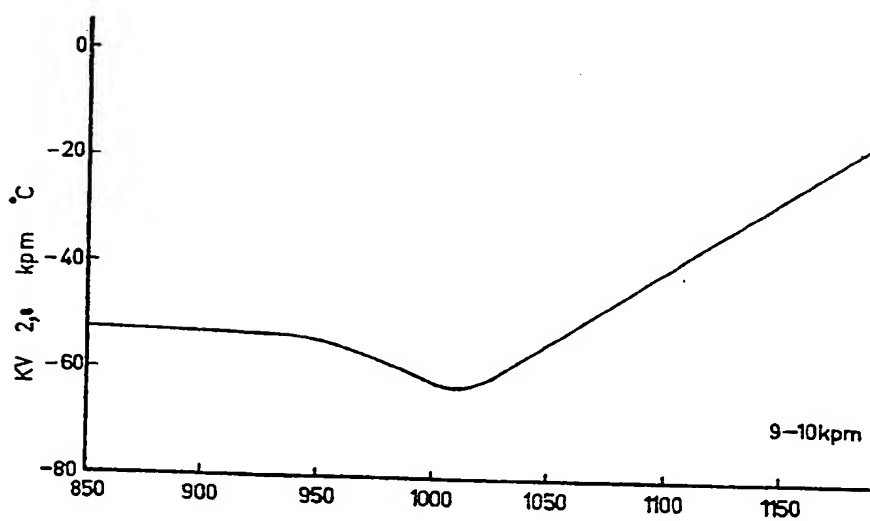


FIG. 2

FIG. 3



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SHEETS 2 & 3

FIG.5

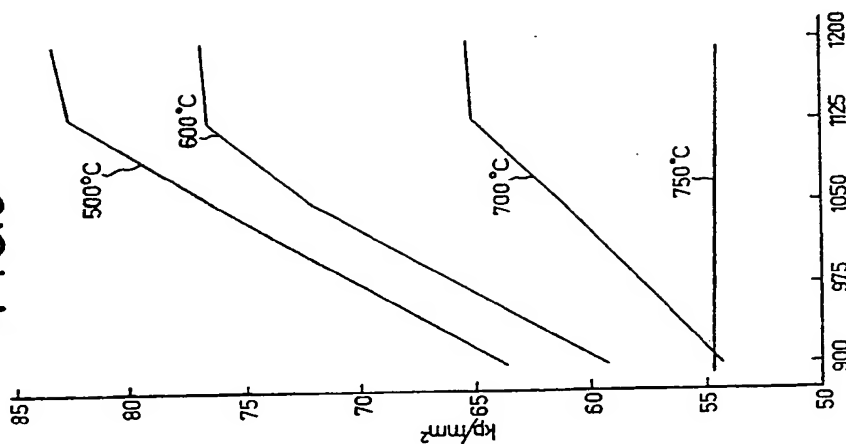


FIG.4

